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Metallic and other inorganic coatings — Review of methods of measurement of thickness

Revêtements métalliques et autres revêtements inorganiques — Revue des méthodes de mesurage de l'épaisseur

Member bodies are requested to consult relevant national interests in ISO/TC 35/SC 9 before casting their ballot to the e-Balloting application.

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 262, *Metallic and other inorganic coatings, including for corrosion protection and corrosion testing of metals and alloys*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 3882:2003), which has been technically revised.

The main changes are as follows:

— editorial revisions;

— restructuring of the document;

— former Tables 2 and 3 moved to Annex A;

— new subclause 6.5.1.2 for the STEP method;

— review of measurement uncertainties;

— phase-sensitive eddy current, as described in ISO 21968, added to measurement methods and Tables A.1 and A.2.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document summarizes the various methods used for the measurement of coating thickness and describes their working principles. Methods of measuring coating thickness are either destructive or non-destructive (see Table 1). The information given in Annex A, Table A.1 will assist in the choice of typical instrumental methods suitable for thickness measurements. For all instrumental methods, manufacturers' instructions contain useful information on the correct handling of the instruments.

The thickness ranges covered by the different methods depend on the coating materials, thickness of the coating, substrates and instruments used (see Annex A, Table A.2); for example, although X-ray spectrometry can be used to measure the thickness of a chromium coating, thicknesses of 20 µm or more cannot be measured with sufficient precision. Similarly, while magnetic methods can be used to measure the thickness of a gold coating over a magnetic steel substrate, many magnetic instruments do not have the sensitivity to measure accurately thicknesses of gold coatings less than 2 µm.

Where a referee method is required, the appropriate coating specification can contain useful information on the preferred method.

Metallic and other inorganic coatings — Review of methods of measurement of thickness

1 Scope

This document reviews methods for measuring the thickness of metallic and other inorganic coatings on both metallic and non-metallic substrates (see Tables 1, A.1 and A.2). It is limited to tests already specified, or to be specified, in International Standards and excludes certain tests that are used for special applications.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Overview

Table 1 summarizes the methods of measuring coating thickness that are discussed in this document.

Table 1 — Methods of measuring coating thickness

Non-destructive			Destructive	
Split beam microscope (light section)		ISO 2128[a]	Microscopical (optical)	ISO 1463
Magnetic		ISO 2178 and ISO 2361	Fizeau multiple-beam interferometry	ISO 3868[b]
Eddy current	— amplitude-sensitive	ISO 2360	Profilometric (stylus and optical)	ISO 4518[b]
	— phase-sensitive	ISO 21968		
X-ray spectrometric		ISO 3497	Scanning electron microscope	ISO 9220
Beta backscatter		ISO 3543	Dissolution methods:	
			Gravimetric strip and weigh method and gravimetric analytical method	ISO 10111
			Coulometric method	ISO 2177
			STEP method	ISO 16866

[a] Can be destructive in some applications.

[b] Can be non-destructive in some applications.

5 Non-destructive methods

5.1 Split beam microscope (light section) method, ISO 2128

This equipment, originally designed for the measurement of surface roughness, is used for measuring the thickness of transparent and translucent coatings, in particular anodic oxide coatings on aluminium. A light beam is projected on to the surface at an angle of 45°. Part of the beam is reflected from the surface of the coating and the rest penetrates the coating and is reflected from the coating–metal substrate interface. The distance that separates the two images observed in the eyepiece of the microscope is proportional to the thickness of the coating and can be measured by means of a Vernier screw that controls a calibrated graticule. The method can be used where sufficient light is reflected from the coating–metal substrate interface to give a clear image in the microscope. For transparent or translucent coatings, such as anodic oxide films, this method is non-destructive.

For measuring the thickness of opaque coatings, a small area of the coating is removed; in this application, the method is destructive. The step between the surface of the coating and the basis metal produces a deflection of the light beam that gives an absolute measure of the coating thickness.

The method is not suitable for hard anodic coatings or for coatings that are very thin (less than 2 µm thick), very thick (greater than 100 µm thick) or rough. It is not suitable for coatings on heavily shot-blasted surfaces. Other methods, such as eddy current (see ISO 2360), interference microscope (see ISO 3868) and microscopical (see ISO 1463), can be applicable for thickness measurement where the split beam microscope method cannot be used.

The method is best suited to small parts because of the ease with which they can be set up on the microscope stage.

The measurement uncertainty of the method is usually less than 10 % of the thickness.

5.2 Magnetic methods, ISO 2178 and ISO 2361

Instruments for these methods measure either the magnetic attraction between a magnet and the basis metal, as influenced by the presence of the coating (see ISO 2361), or the reluctance of a magnetic flux path passing through the coating and the basis metal (see ISO 2178).

All instruments using magnetic methods are sensitive to the magnetic condition and properties of the test specimen, surface curvature, surface cleanliness, surface roughness and thickness of the basis metal and of the coating. Additionally, different materials can look the same but have different permeability, μ_r. Usually, this effect is determined by calibration at the test specimen.

These methods are limited in practice to non-magnetic coatings on a magnetic substrate (see ISO 2178) and to electroplated nickel coatings on a magnetic or non-magnetic substrate (see ISO 2361).

The measurement uncertainty of the method is less than 10 % of the thickness or 1,5 µm, whichever is the greater.

5.3 Eddy current methods, ISO 2360 and ISO 21968

ISO 2360 describes an amplitude-sensitive method and is based on differences in electrical conductivity between coatings and substrates. The method is used primarily for measuring the thickness of non-conductive coatings on non-magnetic metals and of single layer metal coatings on non-conductors. If this method is used for measuring thicknesses of metallic coatings on metallic substrates, great care is necessary if acceptable results are to be obtained. In the latter case, the phase-sensitive method as described in ISO 21968 is more suitable. The phase-sensitive eddy current method is also more suitable for metallic coatings on non-conductive base materials than the amplitude-sensitive method. Furthermore, within certain limits it can deal with non-magnetic metallic coatings on magnetic base materials and with magnetic coatings on non-conductive or conductive base materials.

The method is ideal for rapid determination of anodic coating thickness measurements on aluminium and its alloys and is well suited for use in field measurements. For autocatalytic nickel coatings, this method gives erratic measurements due to variations in conductivity of the coatings with changes in phosphorous content.

The measurement uncertainty of the method is usually less than 10 % of the thickness or 0,5 µm, whichever is the greater.

5.4 X-ray spectrometric method, ISO 3497

This method uses emission and absorption X-ray spectrometry for determining the mass per unit area of metallic coatings, which, provided that the test specimen is of uniform density, is directly proportional to the thickness. In principle, the method can also determine the composition of an alloy coating. Within its physical limitations, it can also analyse superimposed coatings.

X-rays are made to irradiate a fixed area of the coated surface, and the intensity of the secondary radiation emitted by the coating or by the substrate and attenuated by the coating is measured. A correlation exists between the intensity of the X-rays and the coating thickness. This is either established using calibration standards or calculated by the so-called fundamental parameter method and calibration standards are only needed to ensure traceability and to adjust in some cases the calculated characteristic.

This method's accuracy is reduced:

— when constituents of the coating are present in the basis metal, and vice versa;

— when more than two coatings are superimposed;

— when the chemical composition of a coating varies from that of the calibration standard.

The method is not applicable above a certain thickness that depends on the atomic numbers and densities of the materials involved. It also needs a minimum mass per area to deliver results with acceptably low uncertainty.

For autocatalytic nickel coatings, this method is only recommended for deposits in the as-plated condition. The phosphorus content of the coating should be known in order to enable calculation of the thickness of the deposit. However, instruments working with an evacuated chamber, as well as instruments having an SDD detector and working with short working distances and large collimators or capillary optics, can determine the phosphorous content and measure the thickness of the coating in two consecutive measurements, which run in practice quasi as one measurement, using different acceleration voltages for both tasks. As the matrix effect due to the distribution of phosphorus in layers of the coating also affects the measurement uncertainty, the calibration standards shall be made under the same conditions as those of the production process.

Instruments capable of measuring the thickness of a coating with an uncertainty of less than 10 % of the thickness are commercially available.

5.5 Beta backscatter method, ISO 3543

This method uses radio isotopes that emit beta rays and detectors that measure the intensity of those beta rays backscattered by the test specimen. The intensity of the backscattered beta rays will be between two values, namely the backscatter intensity of the coating and that of the basis metal. The measurement is only possible if the atomic number of the coating material is sufficiently different from that of the substrate. The instrument is calibrated using calibration standards having the same coating and substrate as the specimen to be measured. The measured intensity of the beta rays that are backscattered by the test specimen is used to calculate the mass per unit area of the coating. If the test specimen has a uniform density, this intensity of the backscattered rays is directly proportional to the thickness.

This method can be used for both thin and heavy coatings, the minimum and maximum measurable coating thickness being a function of the coating material density.

A measurement uncertainty of less than 10 % of the measuring values over a wide range of coating thickness can be achieved using this method. Depending on the measuring time, the precision of the measuring values can be increased.

6 Destructive methods

6.1 Microscopical (optical) method, ISO 1463

In this method, coating thickness is measured on a magnified image of a cross-section of the coating using an optical microscope.

The measurement uncertainty of this method is less than 10 % of the thickness subject to a minimum error of 0,8 µm. However, with careful preparation of the specimen and the application of suitable instruments, this method is capable of providing a measurement uncertainty of 0,4 µm under reproducible conditions.

6.2 Fizeau multiple-beam interferometry method, ISO 3868

By completely dissolving a small area of the coating without attacking the substrate, or by masking an area before plating, a step is formed from the surface of the coating to that of its substrate. The height of this step is measured with a multiple-beam interferometer.

This method is particularly useful for measuring the thickness of very thin opaque metal coatings. It is not suitable for vitreous enamel coatings. This method is essentially a laboratory technique and is useful for measuring the thickness of coatings on standards used for calibrating non-destructive test instruments, such as beta backscatter and X-ray spectrometry instruments, especially for standards with rather thin (sub-micron) coatings.

This method provides an absolute measure of the microscopic vertical surface variation ranging from 0,002 µm to 0,2 µm. The measurement uncertainty is ± 0,001 µm.

6.3 Profilometric method, ISO 4518

By masking during the coating process or by dissolving a small area of the coating without attacking the substrate, a step is formed from the surface of the substrate to that of the coating. A stylus is drawn across this step and its height is determined by electronically measuring and recording the motion of the stylus. Alternatively, optical profilometers such as confocal microscopes or interference microscopes can be used to measure the step height.

Available commercial instruments allow measurements to be made over the range 0,01 mm to 0,000 02 mm (20 nm).

The measurement uncertainty of this method is less than 10 % of the thickness.

6.4 Scanning electron microscope method, ISO 9220

In this method, coating thickness is measured on a magnified image of a cross-section of the coating using a scanning electron microscope. The measurement is made on a calibrated digital image provided by the SEM's software.

The measurement uncertainty is less than 10 % of the thickness or 0,1 µm, whichever is the greater.

